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<b>(54) Title:</b> AZEOTROPE AND AZEOTROPE-LIKE COMPOSITIONS OF 1-BROMOPROPANE AND DICHLOROPENTAFLUOROPROPANES  <b>(57) Abstract</b>  A cleaning and degreasing solvent comprising 1-bromopropane and a dichloropentafluoropropane. Preferably the dichloropentafluoropropane consists essentially of a mixture of 3,3-dichloro-1,1, pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane. The cleaning and degreasing solvent optionally includes stabilizers and co-solvents to ameliorate the chemical and physical properties of the cleaning solvents. The stabilizer and co-solvents include alcohols, ketones, ethers, acetals, nitroalkanes, epoxides, amines, saturated hydrocarbons, alkenes, alkynes and esters. The solvent composition of the present invention is useful for cleaning a wide variety of articles having metal, cloth, ceramic and plastic surfaces.		

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## **AZEOTROPE AND AZEOTROPE-LIKE COMPOSITIONS OF 1-BROMOPROPANE AND DICHLOROPENTAFLUOROPROPANES**

### **Reference to Related Application**

This application claims priority to a co-pending United States Provisional Application serial # 60/052,006 filed on July 9, 1997 by Richard J. DeGroot.

### **Field of the Invention**

10        This invention relates to a cleaning solvent comprising a hydrochlorofluorocarbon and 1-bromopropane and to a method of cleaning an article using the cleaning solvent composition. More specifically this invention relates to azeotrope and non-azeotropes cleaning solvents comprising dichloropentafluoropropane and 1-bromopropane and to a method of cleaning an  
15        article having a metal, plastic, ceramic, or cloth surface.

### **Background of the Invention**

      The continued drive toward miniaturization of electronic components and electrical circuit boards force stringent processing requirements. The electronic  
20        circuit boards are becoming more densely populated with increasingly sophisticated and delicate electronic circuitry and microchips. Manufacture of these electronic boards and components often require a multistep process. Residue from a previous process step that will interfere with a subsequent step or the performance of the electronic circuitry must be removed before the circuit board  
25        progresses to the next processing station. It is critical that electronic boards and the included circuitry be thoroughly cleaned after manufacture to prevent the accumulation of solder, flux, flux-residues, and other particles from interfering with the function of the electrical circuitry.

      Hydrochlorofluorocarbons (HCFCs) are widely used as cleaning and  
30        degreasing solvents for electronic boards as well as a wide variety of other products that have cloth, plastic, elastomeric, metallic, and ceramic components or surfaces. The HCFC's have demonstrated capability to clean electrical and

mechanical components. Ideally, the cleaning solvents are non-toxic, environmentally benign, low boiling, non-flammable and inexpensive. In addition, to be effective, the solvents must have high solvating ability to dissolve, solubilize, and entrain the organic residues such as the flux and flux-residues on circuit boards without damaging the circuitry and other electronic components on the circuit boards that are being cleaned. The physical and chemical characteristics of the cleaning solvents can be adjusted by combining the HCFC's with other organic solvents to provide a solvent with sufficient solvating power to clean the component safely, effectively, and at a commercially acceptable cost. While a particular solvent mixture initially may provide the desired characteristics in bulk, i.e. non-toxic, low boiling, inexpensive and high solvating ability, in practice the mixture may be inadequate.

Most solvent mixtures partition during use, especially when the solvent is heated during the cleaning process or during solvent recovery. That is, lower boiling components concentrate in the vapor phase and higher boiling components concentrate in the liquid phase. Partitioning of the mixture provides a solvent that is deficient in one or more of its constituents. The resulting solvent does not have the same properties as the original solvent mixture. For example, solvents are often heated to boiling to vaporize the solvent composition in a vapor defluxing or degreasing system. The vaporized solvent condenses on components such as circuit boards that are inserted into the system. When partitioning occurs, the composition of the vaporized solvent differs from the liquid solvent in the solvent reservoir. Partitioning results in a solution that can detrimentally affect the safety and efficacy of the cleaning operation. It is, therefore, advantageous to provide non-partitioning solvents to ensure safe and effective cleaning and degreasing operations.

It is also advantageous to minimize partitioning during solvent recovery to improve efficiency and decrease costs. Solvents are often recovered by distilling used or contaminated solutions to provide essentially pure solvents that can be reused. Partitioning during recovery requires that one or more of the original

components be added to the recovered solvent to maintain the original solvent composition.

Azeotropic mixtures are combinations of solvents that do not partition; instead, they exhibit characteristics of a single pure compound. Thus, they  
5 maintain a constant composition during evaporation and distillation. The azeotrope mixtures exhibit either maximum or minimum boiling points. These characteristics are important when using solvent compositions to remove solder flux and other organic residues from electronic components or grease and oil from mechanical components. The azeotropic mixtures are particularly desirable in  
10 vapor degreasing operations, where a vaporized solvent is employed as a cleaning solution. Furthermore, azeotrope or azeotrope-like compositions also allow for solvent recovery without the necessity of adding one or more of the components to maintain the desired solvent composition.

## 15 Summary of the Invention

The present invention provides a solvent composition comprising dichloropentafluoropropane and 1-bromopropane. Preferably the dichloropentafluoropropane consists essentially of 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane. Specific  
20 solvent compositions of 1-bromopropane and dichloropentafluoropropane provide azeotrope or azeotrope-like compositions that are particularly useful in vapor degreasing and defluxing operations. Optionally, the solvent composition comprises stabilizers, and co-solvents such as alcohols, ketones, ethers, acetals, nitroalkanes, epoxides, amines, and saturated and unsaturated hydrocarbons.  
25 Azeotrope or azeotrope-like compositions were determined to exist when the dichloropentafluoropropane, 1-bromopropane, stabilizers and co-solvents were combined in specific proportions. Admixture of 1-bromopropane, dichloropentfluoropropane, stabilizers, and co-solvents provides a cleaning solution having a high solvating ability, low boiling point, and low toxicity.

The present invention also provides a method for cleaning articles having cloth, metal, plastic, and ceramic surfaces using the solvent compositions that include dichloropentafluoropropane and 1-bromopropane. Optionally, the solvent composition includes stabilizers and co-solvents to ameliorate the stability and solvating ability of the cleaning solvent. The solvent is applied to the articles such as electronic components, metal, plastic, ceramic articles or textiles by any of the known or conventional methods used to solvate and entrain grease, oils, and other particles adhering to the article's surface. Removal of the contaminated solvent provides a cleaned article that is suitable for subsequent processing or forwarding to the consumer.

**Detailed Description of the Invention**

This invention is directed to a solvent composition comprising dichloropentafluoropropane, and 1-bromopropane that provides a cleaning and degreasing solvent having a high solvating ability, low toxicity, low environmental hazards and low boiling. Preferably, the solvent composition is an azeotrope or azeotrope-like composition of the dichloropentafluoropropane and 1-bromopropane. However since the physical and chemical characteristics of the cleaning solvent can be adjusted or modified by admixing the dichloropentafluoropropane and 1-bromopropane in varying proportions, the solvent composition also comprises concentrations of the dichloropentafluoropropane and 1-bromopropane other than the azeotrope and azeotrope-like composition. Such non-azeotrope solvent compositions are useful when solvent partitioning is not a concern or when the cleaning process or solvent recovery does not partition the solvent.

Optionally, the solvent composition includes one or more stabilizer components and co-solvents such as alcohols, ketones, ethers, acetals, nitroalkanes, epoxides, amines, and saturated and unsaturated hydrocarbons. Preferably, admixture of dichloropentafluoropropane, 1-bromopropane and one or more of the stabilizers and co-solvents provides a solvent composition that is an azeotrope or azeotrope-like composition. The solvent composition prepared in accordance with this invention provides a cleaning and degreasing solvent that is useful for cleaning a wide variety of articles having cloth, metal, ceramic, plastic and elastomeric surfaces.

For the purposes of this invention, an azeotrope composition is defined as a constant boiling liquid admixture of two or more substances that exhibits physical characteristics of a single compound, in that the vapor, produced by partial evaporation or distillation of the liquid, has substantially the same composition as the liquid, i.e., the admixture distills without substantial change in the composition. Constant boiling compositions, which are characterized as azeotropes, exhibit either a maximum or minimum boiling point as compared with that of the non-

azeotropic mixtures of the same substances. The present invention contemplates the admixture of the dichloropentafluoropropane, 1-bromopropane, and optionally one or more stabilizers and co-solvents in an azeotrope mixture.

By azeotrope-like compositions it is meant that a composition of  
5 compounds has a concentration that may vary, however minor, from the concentrations found in the azeotropic compositions. Thus the concentrations of the dichloropentafluoropropane, 1-bromopropane, and added stabilizers and co-solvents included in an azeotrope-like composition may vary somewhat from the concentrations found in the azeotrope formed between them and remain a  
10 composition within the scope of this invention. The boiling points of the azeotrope-like compositions will be substantially the same as those of their corresponding azeotropes. Preferably, the azeotrope-like compositions boil, at ambient pressure, at temperatures that are within about 2°C of the temperatures at which their corresponding azeotropes boil at the same pressure.

15 Furthermore, compositions of dichloropentafluoropropane, 1-bromopropane and additional stabilizers and co-solvents that, when fractionally distilled, provide a distillate that is an azeotrope or an azeotrope-like composition when the concentrations of the dichloropentafluoropropane, 1-bromopropane and added stabilizers and co-solvents are different from the concentrations of the  
20 azeotrope or azeotrope-like compositions are within the scope of this invention. Preferably, the concentrations of the dichloropentafluoropropane, 1-bromopropane, and added stabilizers and co-solvents of such compositions differ from the concentration of the azeotrope or azeotrope-like composition by no more than about 10%, more preferably, no more than about 5% by weight.

25 By solvating ability is meant the characteristic of a composition in the liquid state to dissolve solid or semi-solid matter and become miscible with liquids including gums, greases and gels. On a molecular level, solvation entails dispersing molecules of the matter with solvent molecules. The dissolution and miscibility does not have to be complete, i.e. infinite solubility or miscibility with  
30 the matter. However, it is understood that increasing the amount of the solvent



composition added to the substrate by repeated application of essentially non-contaminated solvent solubilizes more of the matter with each increase in solvent amount or each successive application of essentially non-contaminated solvent.

One method of evaluating a solvent's solvating ability is to measure its Kauri

5 Butanol Value as described in ASTM D1133-94 Standard Test Method for Kauri Butanol Value of Hydrocarbon Solvents.

The solvent composition of the present invention comprises an azeotrope or azeotrope-like composition comprising dichloropentafluoropropane. Preferably the dichloropentafluoropropane consists essentially of 3,3-dichloro-1,1,1,2,2-  
10 pentafluoropropane, 1,3-dichloro-1,1,2,2,3-pentafluoropropane, or a mixture thereof. The dichloropentafluoropropanes are commercially available from a variety of sources. A mixture of 3,3-dichloro-1,1,1,2,2-pentafluoropropane, 1,3-dichloro-1,1,2,2,3-pentafluoropropane in a weight ratio of 45:55 is sold under the trade name AK 225 by Asahi. Dichloropentafluoropropanes are low boiling, non-  
15 toxic, non-flammable, and generally chemically stable to most substrates.

The solvent composition also includes 1-bromopropane, which is commercially available from Great Lakes Chemical Corporation. This halogenated solvent is low boiling, inexpensive, and considered less harmful to the atmospheric ozone layer than chlorofluorocarbons. Importantly, 1-bromopropane is non-toxic,  
20 unlike many other halogenated alkanes; for example, 2-bromopropane is considered to be extremely toxic to both humans and animals. Furthermore, 1-bromopropane has an extremely high solvating ability as is exemplified by its Kauri Butanol Value of 125.

While 1-bromopropane provides an excellent cleaning solvent for metal  
25 components, its high solvating ability limits its application for plastics and elastomers; 1-bromopropane tends to etch or partially dissolve plastic surfaces. Admixtures of 1-bromopropane with less aggressive solvents such as hydrochlorofluorocarbons reduces this detrimental effect on plastics and elastometers while at the same time it increases the solvating ability of the

hydrochlorofluorocarbons. The final solvent blend is highly effective for cleaning a wide variety of articles having metallic, ceramic, cloth, and plastic surfaces.

The solvent composition also comprises non-azeotrope compositions of dichloropentafluoropropane and 1-bromopropane. The non-azeotrope solvent compositions are useful for certain applications where partitioning of the cleaning solvent is not a major consideration. The solvent composition comprises 1-bromopropane and about 1% to about 99% by weight dichloropentafluoropropane, preferably about 10% to about 90% by weight, more preferably about 30% to about 70% by weight dichloropentafluoropropane.

Admixture of 1-bromopropane and dichloropentafluoropropane provides a solvent composition with a high solvating ability. By judicious selection of the solvent composition, a cleaning solution having a solvating ability that is lower than 1-bromopropane yet higher than dichloropentafluoropropane can be prepared. The solvent of the present invention comprises about 1% to about 99% by weight 1-bromopropane, preferably about 10% to about 90% by weight, more preferably about 30% to about 70% by weight 1-bromopropane. The solvent compositions of the present invention provide cleaning solvents that have a Kauri Butanol Value of about 32 to about 124, preferably about 45 to about 105, and more preferably about 65 to about 95.

Optionally, the solvent composition includes stabilizers and co-solvents. These stabilizers and co-solvents are included to modify the physical and chemical characteristics of the solvent composition. The stabilizers are added to inhibit metal induced decomposition of halogenated hydrocarbons. Often reactive metals such as aluminum, magnesium, copper, zinc, iron, titanium, tin and alloys of these metals induce decomposition of halogenated hydrocarbons such as 1-bromopropane and hydrochlorofluorocarbons. Typically, these metals induce hydrolysis and/or dehydrohalogenation of the alkyl halides to provide metal halides, halide salts, and acids as some of the decomposition species. Generation of these decomposition species is harmful to metal components. Metal bromides and some of the metal salts are formed from metal ions abstracted from the metal

surface, and the hydrobromic acid severely corrodes metals further exacerbating the problem. Thus, added stabilizers stabilize halogenated solvents in the liquid and vapor state against deterioration in the presence of these metals, inhibit formation of complexes of the metal and decomposition products of the solvent, and reduce attack upon the metal by the acidic decomposition products of the solvent. Stabilizers that are included as optional components of the solvent composition include alcohols, ketones, ethers, acetals, nitroalkanes, epoxides, amines, and mixtures of these stabilizers.

Examples of alcohols that can be added to the solvent include, but are not limited to: ethyl alcohol, propyl alcohol, isopropyl alcohol, t-butyl alcohol, t-amyl alcohol, sec-butyl alcohol, phenols, e.g. phenol, p-cresol, m-cresol, o-cresol, amino alcohols, e.g. monoethanol amine, diethanol amine, triethanol amine, acetylene alcohols, e.g. methylbutynol, methylpentynol, benzotriazol, and mixtures of alcohols.

Typical ketones useful in the present invention include: acetone, methyl ethyl ketone (MEK), 2-propanone (diethyl ketone), 2-pentanone, 3-pentanone, 2-hexanone, 3-hexanone, 2-heptanone, 3-heptanone, 4-heptanone, 2,6-dimethyl -4-heptanone, 2-methyl-3-heptanone, 2-methyl-2-butanone, 2-methyl-3-pentanone, 2-nonanone, and mixtures of ketones.

Specific examples of ethers that can be added to the stabilized solvent include: diethyl ether, dipropyl ether, dibutyl ether, methyl t-butyl ether, 1,4 dioxane, 1,3 dioxalane, trioxane,  $\gamma$ -butyrolactone, tetrahydrofuran, dialkyl ethers of ethylene glycol, e.g. dimethyl ethylene glycol ether, diethyl ethylene glycol ether, and monoalkyl ethylene glycol ethers sold under the trade name CELLOSOLVE that have from 1 to 10 carbons such as methyl cellosolve, ethyl cellosolve, and isopropyl cellosolve. These ethers are added singly or as mixtures of two or more to the solvent.

Examples of acetals suitable for the present invention include dimethyl and diethyl acetals of the ketones listed above.

Typical nitroalkanes useful in the present invention include: nitromethane, nitroethane, 1-nitropropane, 2-nitropropane, nitrobutane, and mixtures of nitroalkanes.

Specific examples of epoxides useful with the present invention include:  
5 epichlorohydrin, epibromohydrin, propylene oxide, 1,2- butylene oxide, 2,3- butylene oxide, cyclohexene oxide, glycidyl methyl ether, glycidyl methacrylate, pentene oxide, cyclopentene oxide, and cyclohexene oxide. The epoxides are added to the stabilized solvent either singly or as a mixture of two or more.

Non-nucleophilic amines are preferred, and therefore secondary and tertiary  
10 amines are desired. By way of example, amines useful for the present invention include: hexylamine, octylamine, 2-ethylhexylamine, dodecylamine, ethylbutylamine, hexylmethylamine, butyloctylamine, dibutylamine, octadecylmethylamine, triethylamine, tributylamine, diethyloctylamine, tetradecyl- dimethylamine, dibutylamine; diisobutylamine, diisopropylamine, pentylamine, N-  
15 methylmorpholine, isopropylamine, cyclohexylamine, butylamine, isobutylamine, dipropylamine, 2,2,6,6-tetramethylpiperidine, N, N-dimethyl-p-phenylamine, N,N- diethyl-p-phenylamine, diethylamine, aniline, ethylenediamine, propylenediamine, triethylamine, tetraethylenepentamine, benzylamine, dibenzylamine, diphenylamine, and diethylhydroxyamine. These amines are useful either singly or  
20 as a combination of two or more.

Examples of co-solvents include: alkanes, alkenes, alkynes, alcohols, ketones, esters, terpenes, and various aliphatic mixtures including mineral spirits, VM & P Naptha and Stoddard solvents. Many of the stabilizers listed above are also considered to be co-solvents. The co-solvents are added to modify the  
25 solvating ability of the solvent composition. Thus, for example, alcohols and ketones can be added to attenuate the solvating ability of the 1-bromopropane. Co-solvents also provide a higher degree of polarity and hydrogen bonding characteristics to the solvent, which enables the solvent to effectively remove ionic or polar contaminants. Furthermore, these co-solvents are often less expensive

than the dichloropentafluoropropanes, and they reduce the costs associated with preparing and using the solvent blend.

The solvent composition is prepared by the admixture of the 1-bromopropane and a sufficient amount of dichloropentafluoropropane to provide the desired cleaning solvent having a specified concentration or a desired Kauri Butanol Value. The order of addition of the components is not critical for this invention. When desired the stabilizers and co-solvents are added. In addition, minor amounts of surfactants can be included. Typical surfactants useful for the invention include ionic and non-ionic surface active agents, for example, sulfonate salts, phosphate salts, carboxylate salts, fatty acids, alkyl phenols, glycols, esters and amides. Surface active agents also include ionic and non-ionic water displacement compounds such as tetraalkyl ammonium sulfonate, phosphate, carboxylate and bromide salts, aliphatic amino alkanols, fluorinated amino alkanols, and chlorofluorinated amino alkanols. Again the order of addition is not critical for the present invention.

The solvent composition of the present invention is suitable for washing articles having cloth, metal, ceramic, plastic and elastomeric surfaces. The solvent composition may be applied by any method known or commonly used to clean or degrease articles. For example, the surface of the article may be wiped with an absorbent medium containing the solvent composition such as a cloth saturated with the solvent. The article may be submerged or partially submerged in a dip tank. The solvent in a dip tank can be either hot or cold, and the article can be submerged for extended periods of time without inducing decomposition of the solvent. Furthermore, the article, dip tank and related components are not harmed by the process. Alternatively, the solvent can be sprayed onto the article or the article can be cleaned in a vapor degreasing chamber with either liquid or vaporized solvent composition.

When the solvent is applied as a vapor, the solvent is typically heated in a solvent reservoir to vaporize the solvents. The vaporized solvent then condenses on the surface of the article. The condensed solvent solvates or entrain grease, oil,

dirt, and other undesirable particles that are on the article's surface. The contaminated solvent drains into the solvent reservoir carrying the dissolved and entrained material to the reservoir. Since only the solvent is vaporized, the grease, oil, and dirt remain in the reservoir, and the article is continually flushed with non-contaminated solvents.

The following examples further illustrate the present invention and are not intended to be limiting in any manner.

### **Example 1 Dichloropentafluoropropane and 1-Bromopropane Azeotrope**

#### **Composition**

A dichloropentafluoropropane mixture of 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane in a weight ratio of 45 to 55 and a sufficient amount of 1-bromopropane were admixed to provide a 50% by weight dichloropentafluoropropane and 50% by weight 1-bromopropane solvent composition. The solvent composition was placed in a Perkin Elmer Model 151 Annular Still (200 theoretical plate capability) and heated to reflux. After the refluxing solvent had reached equilibrium, three distillate samples were collected at a 10 : 1 reflux to takeoff ratio. The samples were analyzed by gas chromatography to determine the relative concentrations of dichloropentafluoropropane and 1-bromopropane. The results are listed in Table 1.

Table 1

Cut	wt% DCPF	wt % n-PB
Initial	50.0	50
Cut 1	95.0	5
Cut 2	92.0	8
Cut 3	92.8	7.2
Bottoms	32.7	77.3

DCPF = of 3,3-dichloro-1,1,1,2,2-pentafluoropropane  
and 1,3-dichloro-1,1,2,2,3-pentafluoropropane,  
n-PB = 1-bromopropane

5

Analysis of the data presented in Table 1 indicates that an azeotrope or azeotrope-like composition could exist for the dichloropentafluoropropane and 1-bromopropane solvent blend. The azeotropic composition was confirmed by preparing and distilling a solvent blend consisting essentially of 93.0% combined  
10 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane and 7.0% by weight 1-bromopropane. The solvent mixture was heated to reflux and allowed to equilibrate. Distillate fractions of approximately 10% by weight were collected using a 10:1 reflux to takeoff ratio. The head and pot temperatures were read directly to 0.1°C. The results are  
15 tabulated in Table 2. Analysis of the data in Table 2 indicates that an azeotrope composition exists. The azeotrope composition consists essentially of about 92% by weight combined 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane and about 8% by weight 1-bromopropane and has a boiling point of about 53°C.

20

Table 2

Cut #	wt g.	wt % Distilled	wt % DCPF	wt % n-PB
Initial	100.7	0.0	93.0	7.7
Forerun	5.4		89.9	7.7
1	11.0	12	98.3	1.7
2	10.7	23	98.6	1.7
3	9.8	33	97.2	2.5
4	20.5	55	96.0	3.1
5	12.9	68	94.0	4.7
6	7.3	76	86.3	11.2
7		76	91.9	5.7
Bottoms	5	81	40.5	56
Average(Cuts 1-4)			97.5	2.3

DCPF = of 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane, n-PB = 1-bromopropane

**Example 2. Solvent Blend Comprising Dichloropentafluoropropane, 1-Bromopropane, and Nitromethane**

A solvent composition comprising about 33% by weight combined 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane (45:55 wt ratio), about 33% by weight 1-bromopropane, and about 33% by weight nitromethane was prepared. Approximately 100 g of the resulting solvent was initially distilled in a Perkin Elmer Model 151 Annular to determine if a ternary azeotrope could exist. Three 10 g fractions from this initial distillation were collected at a 10 : 1 reflux to takeoff ratio. Based upon these preliminary results, a solvent blend consisting of 75.3 g combined 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane, 1.2 g nitromethane and 26.3 g 1-bromopropane was prepared. This solvent blend was distilled in a Perkin Elmer still as described in Example 1. The solvent blend was heated to reflux and allowed to equilibrate. Distillate fractions of approximately 10% by weight were collected, using a 10 : 1 reflux to takeoff ratio. The head and pot temperatures were read directly to 0.1°C. The results from this distillation are tabulated in Table 3.



Table 3

Cut #	wt (g)	wt % Distilled	wt % DCPF	wt % nitromethane	wt % n-PB
Initial	102.8	0	75.3	1.2	26.3
1	10.6	10	91.5		
2	10.8	21	87.7	0.03	6.8
3	6.5	27	89.1	0.03	6.4
4	14.1	41	88.0	6.7	0.03
5	10.7	51	85.5	0.22	12.3
6	10.6	62	77.8	0.42	15.8
7	20.5	82	69.2	1.0	25.5
Bottoms		82	18.0	4.1	75.0

DCPF = 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane, n-PB = 1-bromopropane

5

Analysis of the results tabulated in Table 3 does not confirm the existence of an azeotrope or azeotrope-like composition consisting of 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane, 1-bromopropane, and nitromethane. However, the dichloropentafluoropropane, nitromethane and 1-bromopropane co-distill to provide a solvent blend that does not significantly concentrate one or more constituents in either the liquid or vapor state.

15

**Example 3. Solvent Blend Comprising 3,3-Dichloro-1,1,1,2,2-Pentafluoropropane and 1,3-Dichloro-1,1,2,2,3-Pentafluoropropane, 1-Bromopropane, and Methanol As An Azeotrope Composition**

A solvent composition comprising 33% by weight 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane (45:55 wt ratio), 33% by weight 1-bromopropane and 33% by weight methanol was prepared. Distillation of this initial solvent blend was performed as described in Example 1. Based upon the results of the initial distillation, a second solvent blend comprising

20

62 g of combined 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane, 28.3 g 1-bromopropane, and 11.8 g of methanol was prepared. This solvent blend was distilled in a Perkin Elmer Model 151 Annular Still. The solvent mixture was heated to reflux and allowed to equilibrate.

- 5 Distillate fractions of approximately 10% by weight were collected, using a 10:1 reflux to takeoff ratio. The head and pot temperatures were read directly to 0.1°C. Results from this distillation are listed in Table 4. Analysis of the results in Table 4 reveals that an azeotrope composition exists between 3,3-dichloro-1,1,1,2,2-pentafluoropropane, 1,3-dichloro-1,1,2,2,3-pentafluoropropane, 1-bromopropane and methanol. This azeotrope composition consists essentially of about 76.9% 3,3-dichloro-1,1,1,2,2-pentafluoropropane combined and 1,3-dichloro-1,1,2,2,3-pentafluoropropane, about 7.2% by weight 1-bromopropane, and about 11.7% by weight methanol and boils at about 47°C.

15

Table 4

Cut #	wt (g) Distillate	wt % Distillate	wt % DCPF	wt % Methanol	wt % n-PB
Initial	102.1	0	62.0	11.8	28.3
Forerun	4.8		59.2	11.2	29.6
1	9.3	10	80.4	9.3	6.9
2	6.9	17	76.8	11.3	7.2
3		17	77.2	11.3	7.1
4	8.2	25	73.1	14.8	7.7
5	8.9	34	67.8	16.7	8.0
6	9.7	44	63.3	23.0	9.1
7		44	57.5	28.5	10.3
8		44	21.8	19.7	54.4
Bottoms					
Average	(Cuts 1-4)		76.9	11.7	7.2

DCPF = of 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane, n-PB = 1-bromopropane

**Example 4. Azetrope Composition Consisting of 3,3-Dichloro-1,1,1,2,2-Pentafluoropropane and 1,3-Dichloro-1,1,2,2,3-Pentafluoropropane, 1-Bromopropane and Acetone**

A solvent blend comprising 33% by weight combined 3,3-dichloro-  
5 1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane (45:55  
wt ratio), 33% by weight 1-bromopropane, and 33% by weight acetone was  
prepared and distilled in a Perkin Elmer Model 151 Annular Still as described in  
Example 1. Based upon the results from this initial distillation, a second solvent  
blend comprising 25.7 g combined 3,3-dichloro-1,1,1,2,2-pentafluoropropane and  
10 1,3-dichloro-1,1,2,2,3-pentafluoropropane, 27.0 g 1-bromopropane, and 48 g  
acetone was prepared. This solvent blend was distilled in a Perkin Elmer Model  
151 Annular. The solvent blend was heated to reflux and allowed to equilibrate.  
Distillate fractions of approximately 10% by weight were collected, using a 10:1  
reflux to takeoff ratio. The head and pot temperatures were read directly to 0.1°C.  
15 The results from this distillation are tabulated in Table 5. Analysis of the results  
indicates that an azeotrope exists consisting essentially of about 14.4% combined  
3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-  
pentafluoropropane, about 22.5% by weight 1-bromopropane, and about 65.6% by  
weight acetone, and having a boiling point of about 56°C.

Table 5

Cut #	wt (g) Distillate	wt % Distillate	wt % DCPF	wt % Acetone	wt % n-PB
Initial	100.7	0	25.7	48.0	27.0
Forerun	2.8		26.7	47.6	27.6
1	11.7	20	12.5	73.5	23.1
2	11.7	20	12.5	73.5	23.1
3	11.3	31	14.1	65.9	22.2
4	11.7	43	19.6	60.4	24.2
5	10.7	54	24.2	53.6	24.9
6	11.5	66	30.4	45.9	25.8
7	7.9	74	35.6	39.5	26.4
Bottoms	24.6	99	37.2	22.1	42.7

Average (Cuts 1-4) 14.4 65.6 22.5

DCPF = of 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane, n-PB = 1-bromopropane

What is claimed is:

1. A solvent composition comprising a dichloropentafluoropropane consisting essentially of a mixture of 3,3-dichloro-1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane and 1-bromopropane.
- 5 2. The composition of claim 1 comprising about 10% to about 90% by weight of the dichloropentafluoropropane.
3. The composition of claim 2 comprising about 30% to about 70% by weight of the dichloropentafluoropropane.
4. The composition of claim 1 comprising an azeotrope or azeotrope-  
10 like composition of 1-bromopropane and the dichloropentafluoropropane.
5. The composition of claim 1 consisting essentially of dichloropentafluoropropane and 1-bromopropane that, when fractionally distilled, provides a fractional distillate that is an azeotrope or azeotrope-like composition wherein the concentrations of the dichloropentafluoropropane and 1-bromopropane  
15 differ from the concentrations of the azeotrope or azeotrope-like composition by no more than about 10% by weight.
6. The composition of claim 1 comprising about 1% to about 12% by weight 1-bromopropane.
7. The composition of claim 1 further comprising a stabilizer selected  
20 from the group consisting of alcohols, ketones, acetals, nitroalkanes, epoxides, amines, ethers and mixtures thereof.
8. The composition of claim 7 wherein the stabilizer is a C<sub>1</sub> to C<sub>5</sub> alcohol.
9. The composition of claim 8 comprising a ternary azeotrope or  
25 azeotrope-like composition of the dichloropentafluoropropane, 1-bromopropane, and the alcohol.
10. The composition of claim 8 comprising about 56% to about 81% by weight dichloropentafluoropropane, about 6% to about 11% by weight 1-bromopropane, and about 9% to about 29% by weight methanol.

11. The composition of claim 8 consisting essentially of dichloropentafluoropropane, 1-bromopropane, and methanol that, when fractionally distilled, provides a fractional distillate that is an azeotrope or azeotrope-like composition wherein the concentrations of the
- 5 dichloropentafluoropropane, 1-bromopropane, and methanol differ from the concentrations of the azeotrope or azeotrope-like composition by no more than about 10% by weight.
12. The composition of claim 7 wherein the stabilizer is a C<sub>3</sub> to C<sub>9</sub> ketone.
- 10 13. The composition of claim 12 comprising a ternary azeotrope or azeotrope-like composition of the dichloropentafluoropropane, 1-bromopropane, and the ketone.
14. The composition of claim 12 comprising about 11% to about 36% by weight dichloropentafluoropropane, about 20% to about 27% by weight 1-
- 15 bromopropane, and about 45% to about 74% by weight acetone.
15. The composition of claim 12 consisting essentially of dichloropentafluoropropane, 1-bromopropane, and acetone that, when fractionally distilled, provides a fractional distillate that is an azeotrope or azeotrope-like composition wherein the concentrations of the dichloropentafluoropropane, 1-
- 20 bromopropane, and acetone differ from the concentrations of the azeotrope or azeotrope-like composition by no more than about 10% by weight.
16. The composition of claim 8 wherein the stabilizer comprises a C<sub>1</sub> to C<sub>4</sub> nitroalkane.
17. The composition of claim 16 comprising about 85% to about 92% by weight dichloropentafluoropropane, about 6% to about 16% by weight 1-
- 25 bromopropane, and about 1% to about 7% by weight nitromethane.
18. A method of cleaning an article, said method comprising the step of contacting the article with a solvent composition comprising a dichloropentafluoropropane consisting essentially of a mixture of 3,3-dichloro-

1,1,1,2,2-pentafluoropropane and 1,3-dichloro-1,1,2,2,3-pentafluoropropane having a boiling point of about 54°C and 1-bromopropane.

19. The method of claim 18 wherein the solvent composition comprises about 10% to about 90% by weight of the dichloropentafluoropropane.

5 20. The method of claim 18 wherein the solvent composition comprises about 30% to about 70% by weight of the dichloropentafluoropropane.

21. The method of claim 18 wherein the solvent composition further comprises a stabilizer selected from the group consisting of alcohols, ketones, acetals, nitroalkanes, epoxides, amines, ethers, and mixtures thereof.

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US98/14251

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : C09D 9/00; C11D 7/50; C23G 5/02  
US CL : 510/412; 252/364

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 510/412; 252/364

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS SEARCH TERMS: DECAFLUOROPENTANE, CLEANER, SOLVENT, AZEOTROPE, NON-AZEOTROPE, BROMPROPANE

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,531,916 A (MERCHANT et al) 02 July 1996, col. 5, lines 28-29.	1-4 AND 7,8
Y	US 4,652,389 A (MOLL) 24 March 1987, col. 5, line 54.	1,4,5,6



Further documents are listed in the continuation of Box C.



See patent family annex.

•	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance		
"E"	earlier document published on or after the international filing date	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed	"&"	document member of the same patent family

Date of the actual completion of the international search

24 SEPTEMBER 1998

Date of mailing of the international search report

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Name and mailing address of the ISA/US  
Commissioner of Patents and Trademarks  
Box PCT  
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

SHARON A. GIBSON

Telephone No. (703) 308-0661



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